

REINFORCEMENT OF RICE HUSK IN CERAMIC SHELL MOULD SYSTEM
FOR INVESTMENT CASTING PROCESS

NOOR HASLIZA BINTI KAMARUDIN

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Faculty of Mechanical and Manufacturing Engineering
Universiti Tun Hussein Onn Malaysia

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ABSTRACT

Generally, production of metal component via investment casting process need a development of a very thin ceramic shell mould that can withstand a higher molten metal temperature. Development of ceramic shell mould is very crucial as its involves several times of dipping process followed by the drying and firing stages. Typically, this ceramic shell mould is very brittle and easily prone to cracks that will lead to the casting defects and handling problem at early stage. Therefore, this work focuses on the development of brittle ceramic shell mould via reinforcement method. Rice husk fibers and rice husk ash were selected as a reinforcement material due to the facts that its contains higher percentage of silica. The samples were prepared by three compositions which are untreated rice husk fiber, rice husk treated fiber by sodium hydroxide and rice husk ash. The mechanical and physical properties of ceramic shell mould layering system were measured via Modulus of Rupture, density and porosity, permeability, shrinkage, thermal expansion and observation of metal casting. Experimental results show that the reinforced ceramic shell mould with treated rice husk fiber achieved highest green strength (1.34 MPa). However, ceramic shell mould with rice husk ash shows the highest fired strength (4.87 MPa). Indeed, rice husk fibers additions have greatly increased porosity after firing at 1000 °C by values of 20.06 % and hence promotes increments of permeability by value of $2.46 \times 10^{-11} \text{ cm}^2$. The present of silica also gives highest density, lowest shrinkage and thermal expansion by value of 1.9516 g/cm³, 0.1258 mm and 0.088 % respectively for ceramic shell with rice husk ash. Observation of metal casting shows that reinforced ceramic shell can withstand high molten metal temperature without cracking. Hence, reinforced ceramic shell has reduced production time and cost via avoiding failure to the mould and end cast product.

ABSTRAK

Umumnya, pembuatan komponen logam melalui proses tuangan pelaburan memerlukan acuan cangkerang seramik yang nipis dan mampu menahan suhu lebur logam yang lebih tinggi. Pemprosesan acuan seramik melibatkan pengulangan proses celupan dan diikuti oleh proses pengeringan dan pembakaran. Acuan ini amat rapuh dan mudah retak yang menyebabkan kecacatan pada produk tuangan logam dan menyukarkan pengendalian acuan pada peringkat awal. Oleh itu, kajian ini menumpukan kepada pembangunan acuan seramik melalui kaedah penguatan. Serat dan abu sekam padi dipilih sebagai bahan penguat kerana mengandungi peratusan silika yang tinggi. Penyediaan sampel dibahagikan kepada tiga komposisi iaitu serat sekam padi yang tidak dirawat, serat sekam padi yang dirawat dengan larutan sodium hidroksida dan abu sekam padi. Sifat mekanikal dan fizikal acuan cangkerang dikaji melalui ujian modulus pecah, ketumpatan dan keliangan, kebolehtelapan, pengecutan, pengembangan terma dan pemerhatian tuangan logam. Keputusan eksperimen menunjukkan acuan seramik bertetulang serat sekam padi yang dirawat mencapai kekuatan anum tertinggi (1.34 MPa). Sebaliknya, acuan seramik dengan abu sekam padi menunjukkan kekuatan tertinggi bagi jasad bakar (4.87 MPa). Kesan penambahan serat sekam padi meningkatkan keliangan selepas dibakar pada suhu 1000 °C iaitu 20.06 % dan juga menggalakkan peningkatan kebolehtelapan sebanyak $2.46 \times 10^{-11} \text{ cm}^2$. Kehadiran silika memberi ketumpatan yang tinggi, pengecutan dan pengembangan terma yang rendah dengan nilai 1.9516 g/cm^3 , 0.1258 mm dan 0.088 % bagi cangkerang seramik dengan abu sekam padi. Pemerhatian semasa penuangan logam menunjukkan acuan seramik bertetulang dapat menampung suhu lebur logam tanpa mengalami keretakan. Oleh itu, acuan bertetulang dapat mengurangkan masa dan kos pengeluaran dengan mengelakkan kegagalan bagi acuan dan produk akhir.

TABLE OF CONTENTS

TITLE	i
DECLARATION	ii
DEDICATION	iii
ACKNOWLEDGEMENT	iv
ABSTRACT	v
ABSTRAK	vi
TABLE OF CONTENTS	vii
LIST OF TABLES	xi
LIST OF FIGURES	xii
LIST OF SYMBOLS AND ABBREVIATIONS	xiv
LIST OF APPENDICES	xv
LIST OF PUBLICATIONS	xvi
LIST OF AWARDS	xvii
CHAPTER 1 INTRODUCTION	1
1.1 Research background	1
1.2 Problems statement	2
1.3 Research objectives	3
1.4 Research scopes	3
CHAPTER 2 LITERATURE REVIEW	5
2.1 Introduction	5
2.2 Investment casting process	5
2.3 Shell mould making process	8
2.3.1 Shell mould built up process	8
2.3.2 Drying stage	10
2.3.3 Sintering stage	14
2.4 Shell mould properties	16

2.4.1	Brittleness	16
2.4.2	Permeability	17
2.4.3	Shrinkage	19
2.4.4	Thermal expansion	20
2.5	Strengthening mechanism	21
2.5.1	Reinforcement technique	22
2.5.1.1	Green body	22
2.5.1.2	Fired body	25
2.5.2	Raw material	26
2.5.3	Patented methods	27
2.6	Shell mould materials	27
2.6.1	Shell mould slurry	28
2.6.1.1	Refractory materials	28
2.6.1.2	Binders	29
2.6.2	Rice husk	30
2.6.2.1	Rice husk fiber	30
2.6.2.2	Rice husk ash	31
CHAPTER 3 METHODOLOGY		33
3.1	Introduction	33
3.2	Raw materials	35
3.2.1	Colloidal silica	36
3.2.2	Mulgrain flour	36
3.2.3	Zircon sand and alumina	37
3.3	Samples preparation	38
3.3.1	Rice husk preparation	38
3.3.1.1	Chemical treatment	38
3.3.1.2	Thermal treatment	40
3.3.2	Shell mould making process	40
3.3.2.1	Wax pattern production	40
3.3.2.2	Slurry production	41
3.3.2.3	Shell mould built-up process	43
3.4	Characterization and testing	45



3.4.1	Thermal analysis (TGA)	45
3.4.2	Scanning Electron Microscopy via SEM	46
3.4.3	X-Ray Diffraction (XRD) analysis	47
3.4.4	Modulus of Rupture (MOR) test	48
3.4.5	Density and porosity test	49
3.4.6	Shell permeability	50
3.4.7	Shrinkage measurement	51
3.4.8	Thermal expansion	52
3.4.9	Observation of metal casting	53

CHAPTER 4 RESULTS AND DISCUSSIONS 55

4.1	Introduction	55
4.2	Analysis of surface modification of rice husks	55
4.3	TGA analysis	57
4.4	XRD analysis	60
4.5	SEM observation	63
4.5.1	Fracture mode and fiber-matrix adhesion in the green shell mould	63
4.5.2	Observation of pore structure in fired shell body	68
4.6	Ceramic shell strength via Modulus of Rupture (MOR) test	69
4.6.1	MOR of green body	70
4.6.2	MOR of fired body	72
4.7	Porosity analysis of the green and fired body	74
4.8	Permeability measurements	75
4.9	Analysis of density test	77
4.10	Shrinkage measurement	78
4.11	Thermal expansion analysis	81
4.12	Observation of metal casting process	83

CHAPTER 5 CONCLUSIONS AND RECOMMENDATION 86

5.1	Conclusions	86
5.2	Recommendations	87

REFERENCES	88
APPENDICES	95
VITAE	105



PTTA UTHM
PERPUSTAKAAN TUNKU TUN AMINAH

LIST OF TABLES

2.1	Investment casting flow process	6
2.2	Primary slurry and stucco material composition	9
2.3	Backup slurry and stucco material composition	9
2.4	Permeability measurements of shell mould system that affected by polymer addition	18
2.5	Mean thermal expansion coefficient for commonly used refractory in investment casting	21
2.6	Commonly used refractories in investment casting	28
2.7	Chemical properties of RHA from rice husk	31
3.1	Raw materials of ceramic shell mould formation	35
3.2	Typical properties of colloidal silica	36
3.3	Chemical analysis of mulgrain flour	37
3.4	Size distribution of mulgrain flour	37
3.5	Chemical analysis (%) of zircon sand	37
3.6	Properties of refractory stucco material	38
3.7	Shell build for mould casting	42
3.8	Slurry specification for fiber modified samples	42
4.1	Schematic diagram showing fracture behaviour of the composite under three-point bend test	64
4.2	Fracture mode of ceramic shell system	66



PTTA UTHM
PERPUSTAKAAN TUNJUNGAN AMINAH

LIST OF FIGURES

2.1	Stuccoing process of ceramic shell mould by rainfall sand technique	10
2.2	Drying stage of a ceramic shell body	13
2.3	Sintering mechanism of ceramic body	15
2.4	Comparison of fired permeability for polymer modified and fiber modified shell system	19
2.5	Secondary electron microscopy image showing the green fracture surface of fiber modified ceramic shell	24
3.1	Flow chart of reinforcement ceramic shell mould system process	34
3.2	Chemical treatment process of rice husk fiber	39
3.3	Production of ceramic slurry	42
3.4	Schematic diagram of ceramic shell mould reinforcement	43
3.5	Ceramic shell mould making process	44
3.6	Schematic operation of SEM	47
4.1	Morphology of rice husk fiber under optical camera observation	56
4.2	SEM surface morphology of rice husk at 200x magnification; (a) untreated rice husk fiber, (b) NaOH treated rice husk fiber	56
4.3	Relative mass change of rice husk samples (TGA)	57
4.4	TGA of untreated rice husks fibers	58
4.5	TGA of treated rice husks fibers	58
4.6	XRD analysis of untreated rice husk ash that burnt at 1000 °C (RHU)	60



PT TANA UTAMA
PERPUSTAKAAN TUNKU AMINAH

4.7	XRD analysis of treated rice husk ash that burnt at 1000 °C (RHT)	61
4.8	XRD analysis of rice husk ash that burnt at 600 °C (RHA)	61
4.9	X-Ray diffraction patterns for different shell mould composites systems; (a) SMWF, (b) SMRHU, (c) SMRHT and (d) SMRHA	62
4.10	Morphology of green fracture surface of ceramic shell mould at different magnification; (a) SMWF, (b) SMRHT and (c) SMRHU	65
4.11	Morphology of fiber-matrix adhesion of ceramic shell mould system at different magnification; (a) SMRHT, (b) SMRHU	67
4.12	Microstructure of fired ceramic shell body via backscattered SEM; (a) SMWF, (b) SMRHU, (c) SMRHT, (d) SMRHA	69
4.13	Ceramic shell strength results for green body shell	70
4.14	Ceramic shell strength results for fired body shell	72
4.15	Porosity test data of green and fired body for ceramic shell mould system	74
4.16	Fired permeability of ceramic shell mould system	76
4.17	Density test data of green and fired body for ceramic shell mould system	78
4.18	Green linear shrinkage of ceramic shell body system	79
4.19	Fired linear shrinkage measurements of ceramic shell mould system	80
4.20	Thermal expansion of ceramic shell mould system	82
4.21	Metal casting of ceramic shell mould system; (a) before metal casting, (b) after metal casting	84



LIST OF SYMBOLS AND ABBREVIATIONS

°C	Degree Celsius
Al ₂ O ₃	Alumina
BSE	Backscattered electron
CaO	Calcium oxide
CO	Carbon oxide
CO ₂	Carbon dioxide
Fe ₂ O ₃	Iron oxide
H ₂ SO ₄	Sulphuric acid
HCl	Hydrochloric acid
HNO ₃	Nitric acid
HPC	High performance concrete
K ₂ O	Potassium oxide
MgO	Magnesium oxide
MOR	Modulus of Rupture
Na ₂ O	Sodium oxide
NaOH	Sodium hydroxide
NH ₄ OH	Ammonium hydroxide
OM	Optical microscope
RHA	Rice husk ash
SE	Secondary electrons
SEM	Scanning Electron Microscope
SiO ₂	Silica
SMRHA	Shell mould with rice husk ash
SMRHT	Shell mould with treated rice husk fiber
SMRHU	Shell mould with untreated rice husk fiber
SMWF	Shell mould without fiber
TGA	Thermal Gravimetric Analyzer
VOC	Volatile organic component
XRD	X-Ray Diffraction

LIST OF APPENDICES

APPENDIX	TITLE	PAGE
A	XRD raw data	96
B	Material sheet data	100



PTTA UTHM
PERPUSTAKAAN TUNKU TUN AMINAH

LIST OF PUBLICATIONS

Journals:

- (i) Zawati Harun, Noor Hasliza Kamarudin, Nur Azam Badarulzaman and Md Saidin Wahab. (2011) “Shell Mould Composites with Rice Husk” Key Engineering Materials. Vol. 471-472, pp 922-927.

Proceedings:

- (i) Zawati Harun, Noor Hasliza Kamarudin, Md Saidin Wahab, Ahmad Mujahid Ahmad Zaidi and Nur Azam Badarulzaman. “Shell mould with treated organic fiber”. 25-26 May 2010, National Conference on Advanced Manufacturing and Material Engineering (NAMME).
- (ii) Noor Hasliza Kamarudin, Zawati Harun, Md Saidin Wahab, and Nur Azam Badarulzaman. “Shell mould reinforced with rice husk”. 26-28 November 2010. 4th International Conference on Postgraduate Education (ICPE4).

LIST OF AWARDS

- (i) **Bronze Medal in Malaysia International Technology Expo [MiTE 2010]:**
Zawati Harun, Noor Hasliza Kamarudin, Ahmad Mujahid Ahmad Zaidi, Md. Saidin Wahab, En. Haffidzudin bin Hehsan, Nur Azam bin Badarulzaman, Mohamed Nasrul Mohd Hatta. “Reinforced Shell Mould (treated organic fiber”.



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CHAPTER 1

INTRODUCTION

1.1 Research background

Fabrication of ceramic shell mould in investment casting technique can be described by immersing wax pattern repeatedly in a liquid ceramic slurry followed by applying a layer of a stucco aggregate to the slurry layer and then repeating the process by a number of times. Adequate amount of time is needed between immersions to allow the slurry coat to partially or completely dry on the wax layer [1-2]. Layering coats is repeated until the desired thickness of the mould is achieved. Usually about 4 to 20 ceramic slurry layers are used for the shell mould. For some other uses, about 10 to 18 layering systems are applied which also depends on the application types of metal casting. When sufficient thickness of ceramic has built up on the wax, it is then removed by flash dewaxing process. The completed mould is then fired, providing it with enough strength to withstand the casting process [2-3].

Ceramic shell moulds must exhibit higher green and fired strength to prevent defects in shell making process (handling stage) and also during the casting stage. The strength and integrity of the mould are very important factors in ensuring that the metal part has the proper dimensions. These shell mould characteristics are especially critical for manufacturing high performance components, i.e. super alloy parts used in the aerospace industry. Ceramic shell casting often requires high temperature in the range of 1450 °C to 1750 °C. But, many conventional shell moulds do not exhibit sufficient strength at these temperatures. The moulds become susceptible to bulge and crack when they are filled with the molten metal. This can alter the dimensions of the moulds causing undesirable variations in the cast product.

Cracking also can result in failure of the mould as the molten material runs out of it [3].

Typically, greater strength and dimensional stability are required for ceramic shell moulds used at very high casting temperatures. In that sense, shell moulds are strengthened by wrapping a fibrous reinforcing material around the shell mould as it is being made. The reinforcing material is wrapped in a spiral fashion around the shell mould with enough tensile strength to keep it in places as ceramic layers are applied to the mould built up process to get desired thickness [1-2, 4-5].

One of the reinforcing material in ceramic shell mould is rice husk fiber. It is an organic fiber or known as biodegradable material which is also a non hazardous element. Rice husk not only can reduce the cracking of shell mould problem but also can reduce the pollution emission level that is always associated with the industrial waste problem. Besides, it contains high percentage of silica element which can withstand high melting temperature during casting process. The treatment of rice husk fiber also provides enhanced ceramic green strength due to coarsening and rough surface that creates bonding in this brittle structure of the green shell mould system. Indeed, silica content has a very low thermal expansion coefficient and is very rigid and strong at high temperatures [6].

1.2 Problems statement

For many years, precision investment casting foundries have periodically reported serious casting defects. The shell mould defect is attributed to the ceramic shell properties that are very brittle and highly sensitive to any thermo physical changes which lead to a sudden failure or cracking. These properties not only lead to the appearance of defect in the end cast product but always attribute to the handling problem at the earlier stage in the investment casting industry due to lower green strength. Many conventional shell moulds do not have the strength to withstand high metal casting temperatures and are not suitable for casting large parts due to the bulging defects that also occur in large parts even at lower temperatures. The mould also becomes dimensionally unstable at elevated temperatures and throughout various cooling cycles. Therefore, cracking mechanism will occur when it is filled with the molten metal that results in the failure of the mould. This situation will lead

to the loss of the cast product or existence of the defects in the final product. Basically, this problem can be solved by improving ceramic shell mould property and performance.

1.3 Research objectives

The objectives of this research were focused on:

- i. To study and investigate the effect of reinforcement of rice husk fiber and silica of rice ash to the properties and microstructure of ceramic shell mould.
- ii. To build a reinforced shell mould that can be strengthen the brittle shell mould which lead to the possibility of using fewer shell layers with equivalent ceramic mould thickness and mechanical performance.
- iii. To develop a reinforced shell mould that creates more micro porous structures to help ease the heat permeation process and also avoid miss-run and non-fill of molten metal.
- iv. To develop a reinforced shell mould that can reduce shrinkage mechanism and thermal expansion during metal casting.

1.4 Research scopes

The scopes of this study were focused on:

- i. Literature study on the ceramic shell mould properties and failure mechanism that causes problem in shell making process and shell mould system.
- ii. Sample preparation of ceramic shell mould via reinforcement method and strengthening mechanism by using untreated rice husk fiber, treated rice husk fiber and silica of rice husk.
- iii. Characterization via Scanning Electron Microscope (SEM), Optical Microscope (OM), X-Ray Diffractometer (XRD) and Thermal

Gravimetric Analyzer (TGA), physical properties (density and porosity, shrinkage, permeability, thermal expansion and metal casting), and mechanical properties via Modulus of Rupture (MOR).

- iv. Analysis on the effect of reinforcement to the overall performance of ceramic shell mould comparison with non reinforced shell mould.



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CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This chapter will focus on the details of investment casting process specifically on the ceramic shell build up process. The requirements of ceramic shell mould properties, raw materials and strengthening mechanisms for the ceramic shell mould structure will be discussed. This chapter also will discuss the possible method for the improvement of shell mould performance including reinforcement technique in ceramic shell mould such as the use of natural fibers as reinforcing material. The consolidation process of ceramic shell structure via drying and sintering stage will be presented. Previous studies related to ceramic characteristic and properties will be used as a guideline to conduct an improvement to the shell mould.

2.2 Investment casting process

Investment casting process has been increasingly used to produce components of aerospace and it is widely used to produce a complex shape of metal component such as single crystal turbine blades that can withstand high temperature application [7]. In addition, metals are hard to machine or fabricate so it is a good material for this process. Ceramic shell mould casting allows dimensionally accurate components to be produced in high or low volumes and is a much cheaper alternative to forging or metal turning since the waste material is kept to a minimum. In addition, compared to other processes, this process requires little surface finishing and only minor machining. The advantages of investment casting are excellent surface finish, high

dimensional accuracy, almost any metal can be cast with no flash or parting lines. As a result, this process is one of the most economic methods of forming a wide range of metal components [8].

Investment casting is known as a lost wax casting process [1] that indicates the process start from invested a wax pattern with a refractory material till to the removal of wax in firing stage (to form a mould). Basically, production of an investment casting component using a ceramic shell mould consists of eight stages which are pattern making, pattern assembly, ceramic shell mould production, dewaxing, firing, casting, shell removal, finishing and inspection. All these steps need to be carried out in a proper condition and some of them are very slow in process and need a controlled environment. This is very important to ensure a good production of end cast product. At the same time, it can reduce the loss of the cost that may occur due to the failure of shell mould (at early stage), cracking of mould during pouring (at intermediate of finishing stage). The details of the investment casting process will be briefly presented and explained in Table 2.1.

Table 2.1: Investment casting flow process [9]

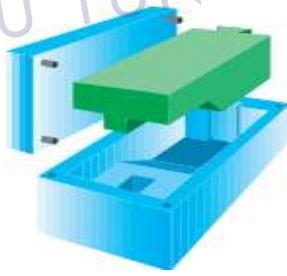

Process	Process Description	Illustration
Pattern making	<ul style="list-style-type: none"> -Wax pattern production by injection molding. -Patterns are made slightly larger to compensate for volumetric shrinkage in the pattern production state and during solidification of metal in the ceramic mould. 	
Pattern assembly	<ul style="list-style-type: none"> -Patterns are fastened on to runners that are attached to the pouring cup. -Patterns, runners and pouring cups comprise the cluster or tree, which is needed to produce the ceramic mould. 	

Table 2.1 (continued)


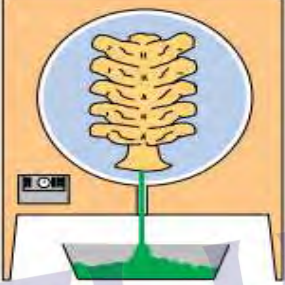
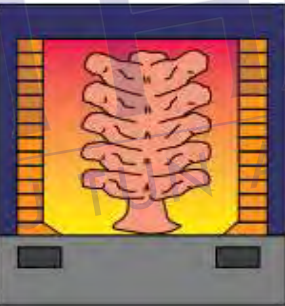
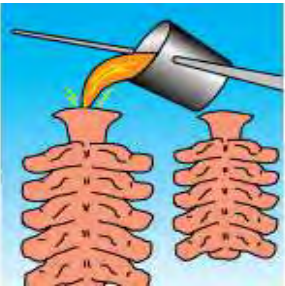
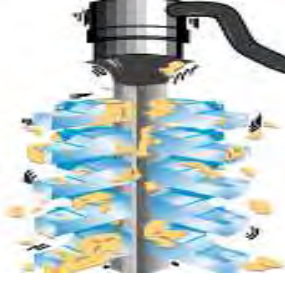
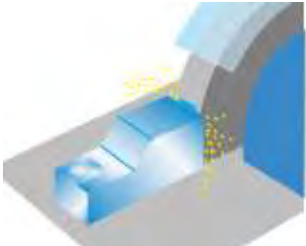
Ceramic shell mould production	<p>-Shell making involves dipping the entire cluster into slurry, draining it, and then coating with fine ceramic sand.</p> <p>-After drying, this process will be repeated until a shell of sufficient thickness has been formed.</p>	
Dewaxing	<p>-It is necessary to remove the wax within the shell by either placing the shell into a steam autoclave or directly into a preheated furnace.</p> <p>-As the wax melts, it exits the shell through the runner or sprue system of the assembly.</p> <p>-After dewax, the shells can be stored in a controlled environment until scheduled to cast.</p>	
Firing	<p>-Prior to casting, the shell is fired to develop the fired strength of the ceramic (green or unfired shells have insufficient strength to contain the metal).</p> <p>-The remaining amount of wax soaked into the ceramic shell is burned out in a furnace.</p>	
Casting	<p>-After proper firing, the shells are removed from the furnace and the moulds are then cast by the molten metal.</p> <p>-It is allowed to cool and solidify into the shape of the final casting.</p>	
Shell removal	<p>-After the poured moulds have cooled, the mould materials are removed from the casting cluster.</p> <p>-This can be done using mechanical vibration, abrasive blasting and chemical cleaning.</p>	

Table 2.1 (continued)

Finishing and inspection	-Finishing operations such as grinding or sandblasting are used to smooth the part at the gates. -Heat treatment is also sometimes used to harden the final part by inspection process.	
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2.3 Shell mould making process

The ceramic shell mould building process involves repeatedly dipping a wax pattern to be cast in ceramic slurry and then stuccoing the slurry layer with stucco material. This process is followed by drying and firing of the layers to produce a shell mould of desired wall thickness on the pattern.

2.3.1 Shell mould built up process

The production of investment casting ceramic shell mould is a crucial part of the whole process and need to be conducted properly to produce maximum shell strength and to avoid other defects in ceramic shell body. It is built from two components which are ceramic slurry and refractory sand stucco. Ceramic shell mould making process starts with the production of ceramic slurries that consist of refractory filler and a binder system (colloidal silica, ethyl silicate or sodium silicate).

The slurry mixing process also has an effect on shell strength and casting quality. Hence, a poorly wet-in slurry will not develop shell with a good and maximum strength [10]. In practice, several slurries are used. First, the shells were made by investing the wax pattern into the primary slurry. The first coating that is applied to the wax pattern is usually the most viscous. The flour that is used in primary slurry composition is finer usually -325 mesh [11]. This ceramic shell layer can produce fine surface finish of casting products and also give sufficient shell strength during casting [2]. The slurry viscosity is measured for several times a day by viscosity cups with a hole in the bottom that is filled with a representative sample

of the slurry. The cup is raised from the slurry and the drain time of the slurry through the hole is measured in seconds [11].

Each succeeding shell layer is built up the same way. However, the backup slurry composition will be different than the primary or first ceramic shell layer. Generally, the backup or secondary slurries are less viscous and the refractory stucco is larger [11]. But, low viscosity slurry will have a greater amount of water and thus will allow more water to soak into the previously dried coats and will soak further back into the shell mould structure. Primary and backup slurries and stucco material composition commonly used in shell formation are shown in Tables 2.2 and 2.3. Moreover, the composition of the slurry and refractory grain is selected primarily based upon the alloys cast [12]. More details of binder and refractory materials of slurry composition will be discussed in the last section of this chapter.

Table 2.2: Primary slurry and stucco material composition [11]

Primary slurry	Primary stucco
1. Binder	-50 to +100 mesh
2. -325 mesh refractory	
3. Wetting agent	
4. Anti-foam agent	

Table 2.3: Backup slurry and stucco material composition [11]

Backup slurry	Backup stucco
1. Binder	-20 to +50 mesh
2. -120 mesh flour	-12 to +30 mesh
3. Solvent	

Then, the stucco or refractory sand is applied onto the wet slurry film to holds the slurry in place and prevents it from running off the pattern. Indeed, it adds strength to the ceramic shell mould [11] (green strength). A dense uniform stucco layer that is applied to uniform slurry will produce the strongest shell. The purpose of stucco is to minimize drying stresses in the coatings by presenting a number of stress concentration centers which distribute, and hence reduce the magnitude of the local drying stress. The second purpose of the stucco is to present a rough surface, thus facilitating a mechanical bond between the primary coating and the back-up or secondary investment [2].

When the primary coat is dried (the binder started to gels), the assembly is systematically dipped into secondary slurry and stuccoes by refractory stucco. This step is repeated until the required thickness of shell is built. This stuccoing process was applied by the rainfall sanding or fluid bed application method [12]. The particle size of the stucco is increased as more coats are added to maintain maximum mould permeability and to provide bulk to the mould. Finally a seal coat of secondary slurry is applied and dried [2]. In shell making process, the bridged or excessive stucco can produce a weak spot that can break and be infiltrated with metal during pouring. This can be prevented by blowing out or shaking off the excessive stucco immediately after the application [14]. Figure 2.1 shows stuccoing process of ceramic shell mould by rainfall sand technique.

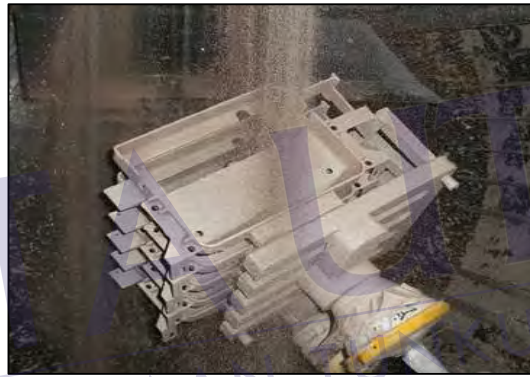


Figure 2.1: Stuccoing process of ceramic shell mould by rainfall sand technique [13]

Thus, an investment casting mould consists of individual layers of fine refractory and granular refractory materials held together by a binder that has been set to a rigid gel. Each coating is thoroughly hardened between dipping [2] via proper shell drying that can establish maximum shell strength and avoid cracking mechanism related to rapid drying rate. Drying mechanism in ceramic shell body in shell making process will be explained in the next section.

2.3.2 Drying stage

Ceramic consolidation occurs when drying and firing is applied to the loose ceramic shell particles. Due to the fact that ceramic is highly thermal sensitive, therefore the

consolidation stage requires to be carried out in systematic way such as drying must be conducted before firing stage [15-16] by removing water content and pore existence. Drying means loss of moisture from the surface of the substance by evaporation. This area has been studied for many years as it is critical in the manufacturing of most ceramic bodies [16]. In investment casting process, drying stage is important for the shell mould green part system due to the high concentration of water in shell mould structure. Basically, shell mould is dried after each dipping and coating process to strengthen the bonds between refractory material layering system [17].

Drying of ceramic shell body is a critical stage and must have moisture removed in a very controlled manner. This is due to the tendency of part to warp or crack from variations in moisture content and thickness within the part and the complexity of its shapes. As a wet slurry coat dries, its volume decreases and the refractory grains are drawn closer to each other. Fortunately, the sequence of dipping process at an interval time allows for much greater flexibility in drying, as the slurry layer is thin and spread over a wide area. In addition, the stucco layer acts to slow down the drying rate on each individual dip by covering the surface of the slurry. Nevertheless, excessive air movement can cause cracking during the first several dipping layering processes due to rapid drying rate. In addition after the coat is dry, a large or rapid temperature change can cause wax expansion which can crack the coating layer [16].

Theoretically, the stresses will occur in the ceramic shell body if the drying rate is too fast. Due to this fact, the cracking mechanism can occurred in ceramic shell bodies if these stresses are large enough. In some instances, although the cracking does not create a major defect, but sometimes it will affect the ceramic bodies such as buckling and lifting etc. When the defect occurs along after the part has been dipped, over drying is usually cited as the cause. The residual stress from rapid drying rate almost creates problem in ceramic bodies and weakens its structure. Hence, drying stage is important in ceramic shell making process and has to be properly conducted. Figures 2.2 (a), (b), (c), and (d) show the stages of drying process of a ceramic body system.

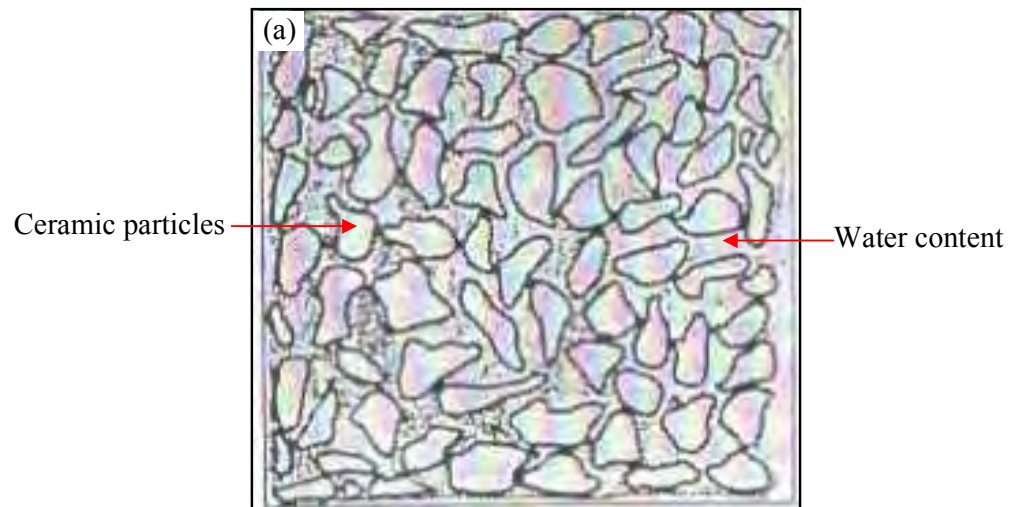


Figure (a) shows a group of particles separated by a large amount of water, which would be the case when a cluster has just been dipped

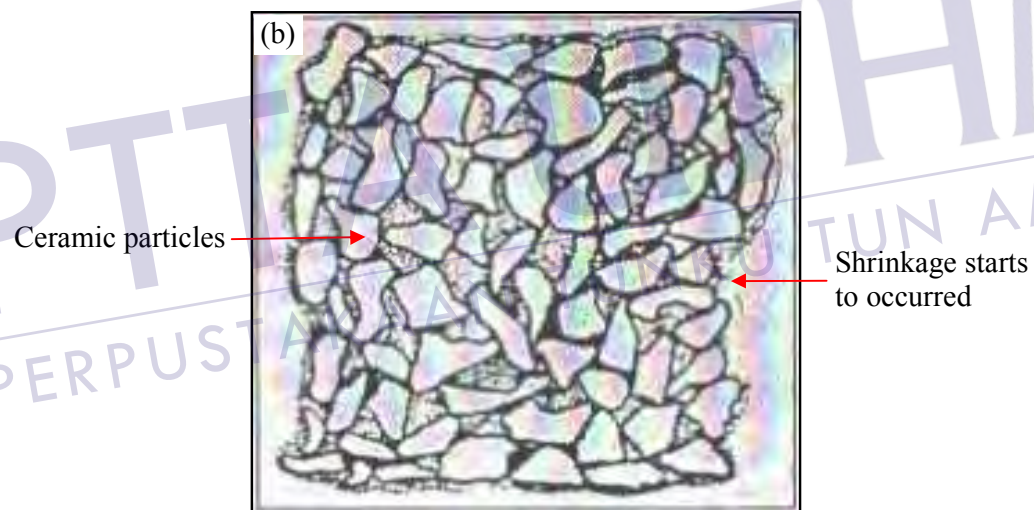


Figure (b) shows some of the water content has been removed after dipping process is finished. Clearly, some shrinkage also has occurred and many of the particles have started to touch to each other.

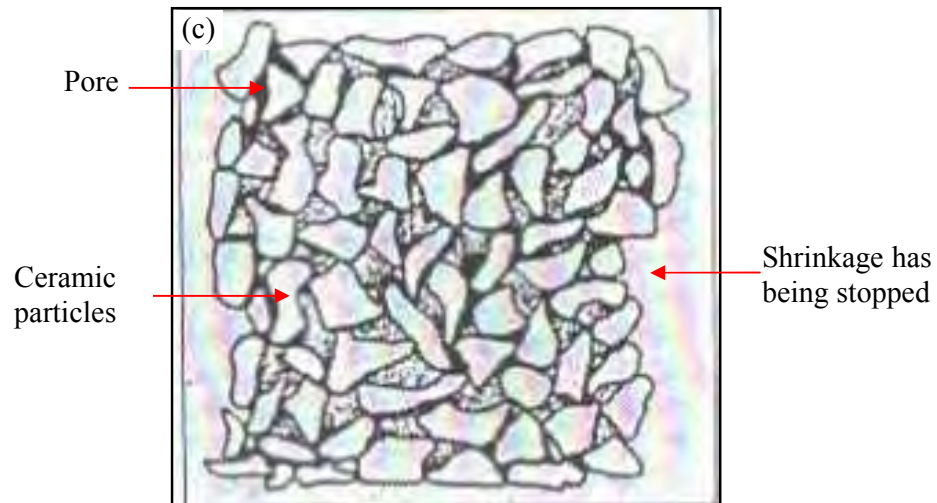


Figure (c) shows drying shrinkage has been stopped and as all the particles have made at least some contact with other ceramic particles. A great deal of water is left in the pores and at inters particle contacts.

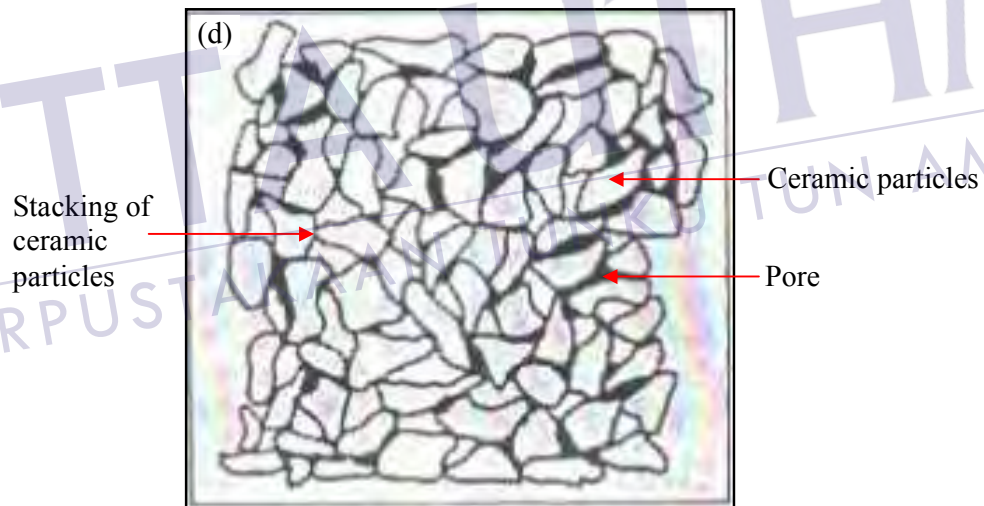


Figure (d) shows that the most of the readily accessible water is gone by water removal during final drying stage. Some liquid in small capillaries remains, along with physically adsorbed water.

Figure 2.2: Drying stage of a ceramic shell body [16]

Colloidal silica binders do not form strong bonding networks when it is formed semi-solid by gellation, which means the slurry layers are bound together [1]. The binder must be set by drying to yield optimum bonding strength. If the colloidal silica binder is not allowed to totally set dried, a certain loss in shell strength will

occur. The amount of the strength loss will depend on how much moisture is left in the ceramic shell body when it is re-dipped or drying time taken [18]. Practically, three hour dry time is enough to achieve optimum bonding as given the same drying conditions. Drying for an additional 24 hours for seal coat layer does not increase the ceramic shell body strength but any residual moisture can be detrimental to the shell during steam de-waxed process due to the large expansion that occurs when water is turned into steam. Indeed, drying for longer times will not increase the strength, but it reduces the potential for autoclave related cracking mechanism [16].

Normally, each layer of the mould takes about one or two hours to produce due to the need for controlled moisture removal in every layer. Unless sufficient moisture is removed, the layer will have insufficient mechanical strength to allow another to be applied. Due to the fact colloidal silica has produced a ceramic shell mould with low green strength, so ceramic green body strength can be enhanced by the addition of organic fiber via reinforcement method along drying stage. So that the drying time of ceramic shell body for two hours interval will be supported by the bonding mechanism of rice husk fiber and ceramic shell matrix. Importantly, this reinforcement can give sufficient green strength (unfired) to withstand wax removal without failure. After drying of ceramic shell body is completed, these samples undergo for the de-waxing followed by the firing.

2.3.3 Sintering stage

After sufficient drying of refractory material layer, the ceramic shell moulds are then allowed for the wax removal followed by the sintering. The final sintering stage in shell formation is undertaken to develop its strength as well as ensuring that the mould is completely dry during removal of water content from the mould. It is done to provide sufficient thermal strength to withstand a high temperature during metal casting process. This is the final step in ceramic shell making process which is one of the most important and crucial part in consolidating ceramic component structure. Firing of refractory ceramic shell mould is actually sintering process which causes the crystals to be combined with one another.

Basically, this stage is ranging from green body forming which starts from the loose particles to the thermal consolidation (drying and sintering stage).

Theoretically, diffusion between each particle during the consolidation in the firing stage leads to the stacking and particle binding together. This will enhance the densification and involves the development of new ceramic structure. This sintering mechanism occurred by diffusion transport of matter along definite path which commonly occur at the boundary of stacked or closed particle. Hence, the strength of the sintered body is increased due to the bonding and growth of necks between the particles [19-21]. The consolidation process of ceramic shell during the sintering binds the particle strongly and prevents the ceramic shell mould from breaking.

The sintering uses a kiln featuring great energy consumption. The green body is dried in the first phase. Normally drying is completed before sintering, but it may contain one to two percent moisture before entering the kiln. Moisture will be evaporated when the temperature is rising. Sintering of ceramic shell involves heating the part to an elevated temperature in a controlled environment, similar to sintering in powder metallurgy. Active sintering takes place during this period, and the ceramic shell body goes on shrinking considerably. Hence, the fired shrinkage not only present problems with the dimensional tolerances of fired ceramic shell body, but it also provides a mechanism through which cracks can be formed.

Sintering at high temperatures causes densification that gives the ceramic shell its strength and other properties. This properties improvement is the result of the development of a strong bond between the complex oxide particles in the ceramic shell bodies. During this process, the individual ceramic particles coalesce to form a continuous solid network and pores are eliminated. Typically, the microstructure of the sintered ceramic shell contains dense grains, where an individual grain is composed of many starting particles. Figure 2.3 shows sintering mechanism in ceramic body.

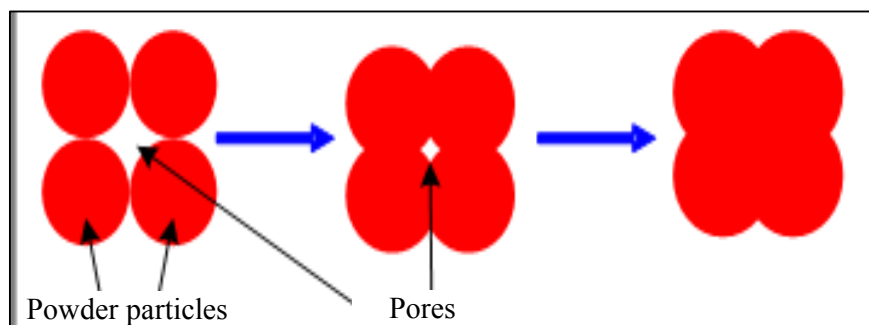


Figure 2.3: Sintering mechanism of ceramic body [13]

2.4 Shell mould properties

In investment casting, a key requirement and ceramic shell mould properties can be described as follows [2]:

- i. Sufficient green strength to withstand wax removal without failure.
- ii. Sufficient fired strength to withstand the weight of the cast metal.
- iii. Sufficiently weak to prevent hot tearing in susceptible alloys.
- iv. High thermal shock resistance to prevent cracking during metal pouring.
- v. High chemical stability.
- vi. Low reactivity with the metals being cast to improve the surface finish.
- vii. Sufficient mould permeability and thermal conductivity to maintain an adequate thermal transfer through the mould wall and ultimately the casting.

2.4.1 Brittleness

Generally, ceramic shell mould has brittle structure that associated with the casting defects. In many applications, brittle fracture limits the use of ceramic materials. In fact, ceramic shell mould has exhibited extreme brittleness and highly susceptible to fractures as it originated from slurry loose particle. As mentioned before, component of slurries are prepared composed of loose particles of fine mesh refractory filler system and a colloidal binder system. This composition produces ceramic mould with very low green strengths which is prone to cracks during wax removal and handling [1-2, 22].

In order to understand the fractural behavior of ceramic materials, it is necessary to understand the mechanisms of ceramic fracture that are entirely brittle. In the ceramic body, plastic deformation by dislocation motion does not occur to such a limited extent that cracks are sharp to the atomic level of the solid. Resistance to a fracture is provided by the lattice itself, and not by the movement of dislocations. Ceramics can be made tougher by modifying the microstructure of the solid in such a way as to reduce stresses near crack tips. The ability to make tougher ceramics has

increased gradually with deepening understanding of brittle fracture [22]. Mechanical behaviors of brittle materials, either static or dynamic, are often dominated by fracture under compression in the presence of confining stresses. At low-stress or no confinement, brittle materials lose their load carrying capacity (shear strength) by axial cracking at some threshold stress (failure threshold) [23].

From the previous study and laboratory work, the most promising candidates for improving ceramic brittleness is continuous fiber ceramic composites via reinforcement method, which has shown high strength and toughness [24]. On the other hand, other related works suggested the strengthening mechanism via reinforcement technique to improve ceramic shell strength performance [1-2, 4]. There are some strengthening mechanisms in this brittle ceramic shell mould body included reinforcement method, raw material and also patented method that will discuss in the section 2.5.

2.4.2 Permeability

The permeability of a material is simply defined as its openness to the transmission of transport phase. This is a method for measuring the interconnected porosity of ceramic shell system. It is especially important to have a permeable shell when there is poor gating. A permeable shell will prevent non-fill of molten metal during casting [18]. Permeability of the shell is the most important factor, with most investment shells having a minimum of 30% open porosity in the structure [25]. Mould fill is improved by increasing the open porosity and hence the permeability of the ceramic shell [2].

Theoretically, the permeability in porous material depends on the porosity distribution and also the material properties itself. Higher permeability is thought to be obtained by controlling the porous microstructure and this is very difficult in ceramic body because they have randomly distributed pores. Thus, preparing porous ceramics which satisfy both high permeability and high mechanical strength is very difficult. These properties are strongly influenced by the microstructure, so that controlling the microstructure of porous ceramics is essential [26].

Commonly, polymer addition not only promotes green strength and elasticity of ceramic shell body system but also at the same time, it has a tendency to promote

a permeable shell system. In a two component system of colloidal slurry and polymer, properties such as strength and permeability can be controlled by adjusting the amount of either component [18]. The use of polymer-modified binder system in shell formation had increased the open porosity due to the burning out of the polymer phase [2]. This in turn increases the permeability of the ceramic shell mould system. Hence, the incidence of miss-run or non fill of the casting product can be reduced. Table 2.4 shows the effect of polymer addition to the permeability of shell mould system.

Table 2.4: Permeability measurements of shell mould system that affected by polymer addition [18]

System	Flow (cc/min)	Permeability
SP-24 % Silica, 0 % Polymer	87	0.000353
SP-24 % Silica, 8 % Polymer	143	0.000538

In other work, the incorporation of nylon fibers in a water based silica binder to replace the costly liquid polymer addition in investment casting mould show that it can increase the shell mould strength. Indeed, the addition of nylon fibers also dramatically increases the fired permeability by a factor of three at casting temperatures, mainly due to increased porosity networks left after the fiber burns out. This is of great benefit to the casting process, allowing increased transport of air displaced from the mould cavity and reducing the defects associated with air entrapment. These results suggest that the fiber system will produce a ceramic shell mould with greatly increased fired permeability when compared with the polymer-modified system making it an excellent alternative [1-2]. Figure 2.4 shows the comparison of fired permeability for polymer modified and fiber modified shell system.

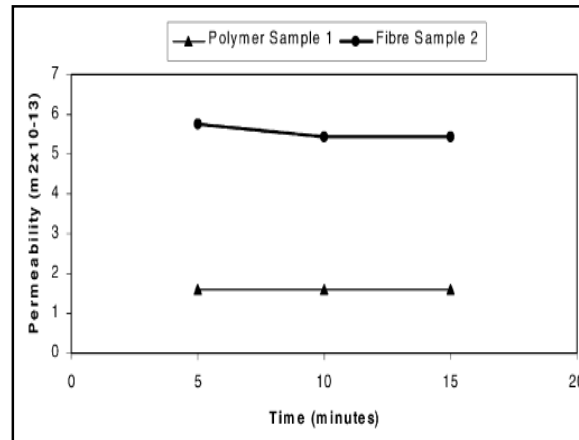


Figure 2.4: Comparison of fired permeability for polymer modified and fiber modified shell system [1-2]

2.4.3 Shrinkage

Shrinkage is the reduction in volume or linear dimensions caused by curing as well as thermal contraction. Dimensional changes suffered by the porous structure during drying are due to the stresses derived from temperature and moisture gradients existing within the body and from capillary forces. Ceramic shell body will shrink once as it dries and then again as it is fired. Firing and drying times also may lead to the variation of shrinkage in every layer of the shell that has a different orientation. Hence, this will be a factor that contribute to cracks during the shell building process [16].

Drying of ceramic component is a complex process involving simultaneously mass and heat transfer [27] that contributes to the loss of moisture from the pore structure. Typically, this process is accompanied by physical and structural changes that are called shrinkage mechanism. Theoretically, the movement of moisture in ceramic body is strongly related to the shrinkage mechanism which occur due to the non-linear conditions [27]. Thus, an excess of moisture movement will result in the uncontrolled shrinkage that will lead to the crack formation and contribute to the failure of material. This phenomenon should be avoided for the highly brittle and thin ceramic body, especially for the case of ceramic shell mould layer system.

Other investigations have shown the use of short randomly distributed fibers [33-34] to significantly limit the effects of shrinkage cracking. The addition of fibers

increases material toughness necessitating increased energy to propagate a crack formation. The addition of fibers is often reported in previous work to have significant reduction in the free shrinkage of paste and mortar specimens via reinforcement technique [35]. Indeed, the addition of micro fine particles of rice husk ash to the concrete mixture would increase the drying shrinkage whereas coarse particles of rice husk ash exhibit lower values than the plain cement based concrete [36].

2.4.4 Thermal expansion

Thermal energy is present and responsible for the atomic or molecular vibration of a mean position in any material. As the temperature of the material is increased, the amplitude of thermal energy induced vibrations is increased and the inter atomic or intermolecular spacing is increased causing an expansion of the body. Most materials show the body expansion with increasing temperature. Over a certain range of temperature, one can relate the temperature interval and thermal strain by a coefficient, called the coefficient of thermal expansion. Thermal stresses are internal stresses that arise when there a constraint on free dimensional change of a body. Thermal stresses are very important in all composite materials but particularly so in ceramic matrix composites because in general, a ceramic matrix has a very low strain to fracture. Ceramics in general also have a lower coefficient of thermal expansion than polymers and metals [37].

Failure due to the thermal shock constitutes a major problem in many applications of refractory ceramics. A significant factor in this troublesome behavior is the high thermal expansion of the materials constituents. In previous work, a fine grained and high density composites structure can promote the highly refractory materials resistant to thermal shocks. The composite product show the advantage of a spectacularly low thermal expansion due to the greater densifications that could probably be attained by sintering at the higher temperatures [38].

Commonly, ceramics shell bodies are brittle and cannot tolerate the sudden changes in temperature (without cracking) if their expansion is too high. The thermal expansion of ceramic bodies can be controlled by firing process to create crystalline bodies that will influence the overall expansion of the material in the desired

direction. Despite of that, thermal expansion generally decreases with increasing bond energy, which also has an effect on the hardness of solids ceramic body. Moreover, harder materials are more likely to have lower thermal expansion. Ceramics body need to be joined or work in consort with a wide range of materials and therefore their expansion must be matched to the application. The dimensional changes can put enough stress on a shell which will cause it to crack [11].

Thermal expansion is important to know when initially setting up a shell system, or switching refractoriness. The dimensions of the cast part may change if a large portion of the shell is changed to a refractory with a significantly different expansion. In addition, other shell problems can occur due to the mismatch in expansion such as cracking mechanism. Table 2.5 shows the mean thermal expansion coefficient for commonly refractory materials used in investment casting. Over the past few years, industry has increased its efforts to introduce new materials for high performance applications [39].

Table 2.5: Mean thermal expansion coefficient for commonly used refractory in investment casting [39]

Material	Linear expansion coefficient (0-1000 °C) in./in. °C x 10 ⁶
Alumina	8.8
Fused silica	0.5
Mullite	5.3
Zircon	4.2
Zirconia (stabilized)	10.0

2.5 Strengthening mechanism

Strengthening mechanism has to be properly conducted to overcome the brittle properties of ceramic shell and it is important to avoid defects in the shell making process and other defects in casting process. This strengthening involves the bonding mechanism and densification of ceramic shell body. The bonding mechanism of ceramic shell mould body appears to be completely different for the unfired and the fired shell mould. The green strength is an indication of ceramic shell strength to be able to withstand the handling stage and de-waxing process. Whereas, fired strength is measured to determine the ability of shell to hold molten metal during casting

without getting cracked and creating other defects. The common methods in strengthening ceramic shell mould are reinforcement technique, raw material and patented methods.

2.5.1 Reinforcement technique

Reinforcement technique is used to strengthen the brittle ceramic structure. Ceramic reinforcement can be produced in the form of continuous fibers, short fibers, whiskers or particles. The incorporation of fibers, whiskers or particles in a ceramic matrix either in green or fired body can result in toughening of brittle ceramic body [40]. This happens due to the incorporation of fiber reinforcements that introduce energy dissipating phenomena such as debonding at the fiber-matrix interface, crack deflection, fiber bridging, fiber pull out, etc. In this regard, proper control of the interface region characteristics in ceramic shell matrix is obviously important [37]. The basic mechanism of strengthening in fiber composite is load transfer by the matrix to the fibers through shear [41]. This reinforcement technique can be applied in the green and fired ceramic shell mould body system to improve the strength performance of a brittle ceramic structure.

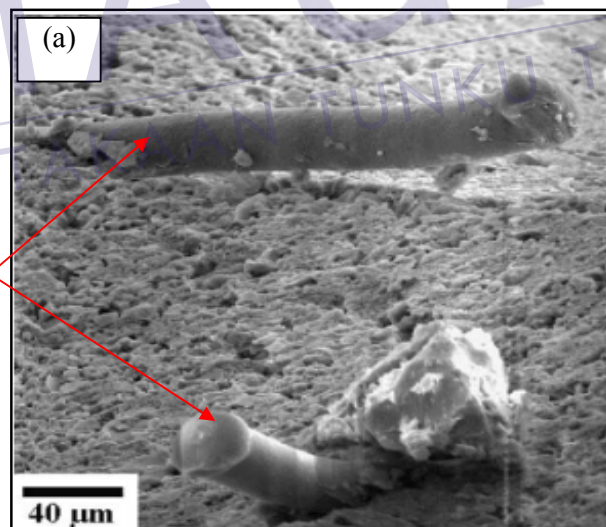
2.5.1.1 Green body

Green ceramic shell strength (before firing) was found to be mostly a function of binder sol characteristics and to be independent of the chemical composition of the bonded particles. Therefore, the handling of this weak green shell mould is always associated with the casting failure problem at the early stage. Basically, the strength of the unfired shell mould is controlled by the amount of binder silica in slurry, alkali content of the binder silica sol and surface area of the bonded particles. The factors that influence the strength of this green ceramic shell moulds are the refractory particles bonded with air dried colloidal silica [16].

One of the methods used for strengthening the nature of brittle ceramic shell mould in investment casting process is incorporating organic fibers into the slurry. In practice, these fibers are designed to act as a composite reinforcing agent that will increase the green strength to prevent cracking within the ceramic structure during

autoclave wax removal [1-2, 4]. In the previous work, strengthening properties of an investment casting mould can be enhanced by nylon fibers additions which show promising increment in ceramic shell green strength performance. The result also indicates that fiber can be used to produce an equivalent ceramic thickness with fewer coats. Ceramic shell would be thicker at the sharp corners providing enhanced mechanical strength at the point of prevalent failure. Thus, an improved performance of ceramic shell mould can be achieved with significant reduction in production time and material cost for investment casting industry.

The smooth nature of nylon fiber surface appears to result in a very easy fiber pulls out from the ceramic matrix during the application of mechanical force. With little resistance there will be little extra energy expended for fracture and the ceramic shell mould will be remaining weak. Hence, fiber addition produces a shell system with slightly lower in green strength [1-2]. Figures 2.5 (a) and (b) show secondary electron microscopy image showing the green fracture surface of fiber modified ceramic shell; (a) fibers pulling out from the surface, (b) the fiber cavity after pull out.



Fibers pulling out from the surface

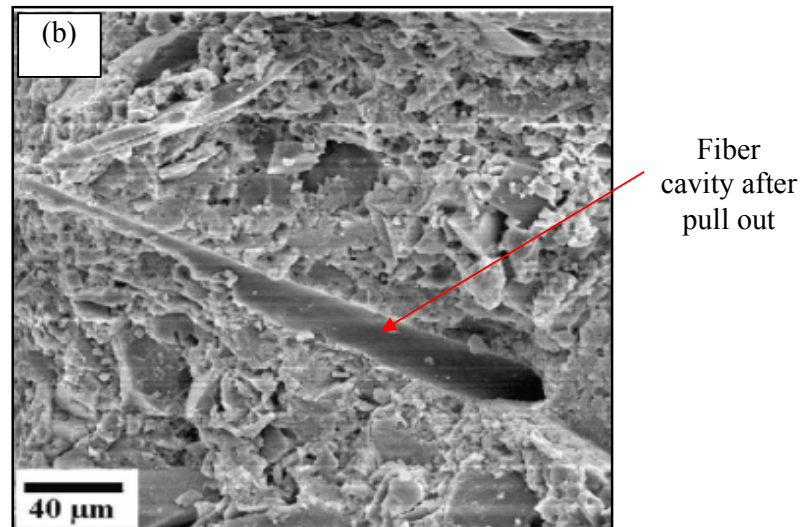


Figure 2.5: Secondary electron microscopy image showing the green fracture surface of fiber modified ceramic shell [1]

In fact, nylon fiber has smooth surface [1-2] so that fiber chemical treatment has been conducted to produced fibers with much rougher surfaces that will allow for mechanical interlocking [37] in ceramic shell body. The interlocking mechanism is mainly influenced by the interfacial adhesion between the matrix and the fibers. Alkaline treatments can improve the tensile and impact properties which appear to be good fiber-matrix adhesion and increased matrix crystallinity. Fiber chemical modifications are employed to improve the interfacial matrix-fiber bonding resulting in the enhancement of strength properties of the composites [42]. It is expected to increase the friction between fiber and shell matrix. Hence, the energy required for fiber pull-out is increased and ultimately increasing the green strength of ceramic shell via reinforcement technique.

Rice husk ash (RHA) are being used to produce more homogenous hydration products by filling and segmentation of the capillary voids and producing ultimately more denser and impermeable concrete. The total porosity is decreased as the percentage of RHA is increased [43]. In other laboratory work found that there are no cracks in concretes made with 15 - 30 % RHA after a few days of exposure. Indeed, it is found that it can slow down the cracking formation in concretes that made with 0 – 10 % of rice husk ash. The replacement of rice husk ash refined the pore and thereby improve strength and corrosion resistance properties [44].

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PTTAUTHM
PERPUSTAKAAN TUNKU TUN AMINAH